

# Davisil® Media

- High surface area
- Tight particle size distribution
- Wide range of pore sizes (30–2500Å) and particle sizes (10–2000µm) available
- Relied on for over 25 years

Recognized worldwide as media of high purity, Davisil® silica is the cornerstone of Grace's offering of silica media products. Consisting of irregularly shaped particles with excellent mechanical and structural properties, Davisil® silicas are versatile, consistent, and reliable. They are ideal for HPLC, solid phase extraction, flash chromatography, and scale-up to industrial process applications.

## Higher Performance

Davisil® silica's chemical and structural properties are optimized for chromatographic performance. Tight control of these properties from raw material to finished product distinguishes Davisil® silica from the competition.\*

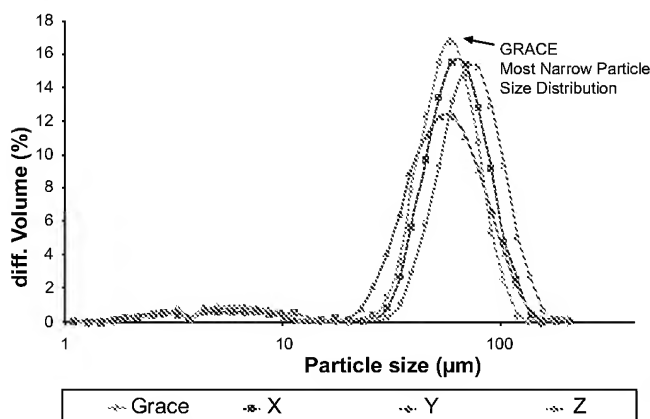
### High Surface Area for Increased Loading Capacity

Company	Surface Area	Bulk Density	Surface Area of 1L Column
Grace	550m <sup>2</sup> /g	420g/L	231,000m <sup>2</sup> /L
X	515m <sup>2</sup> /g	430g/L	221,450m <sup>2</sup> /L
Y	460m <sup>2</sup> /g	430g/L	197,800m <sup>2</sup> /L
Z	450m <sup>2</sup> /g	450g/L	189,000m <sup>2</sup> /L

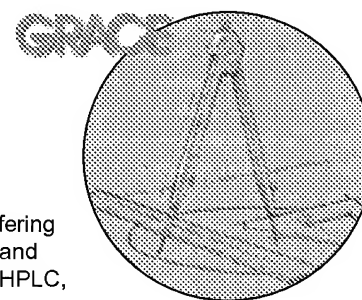
### High Purity Silica to Reduce Unwanted Interactions and Contamination

Company	Mg	Ca
Grace	25ppm	19ppm
X	27ppm	207ppm
Y	119ppm	793ppm
Z	212ppm	1775ppm

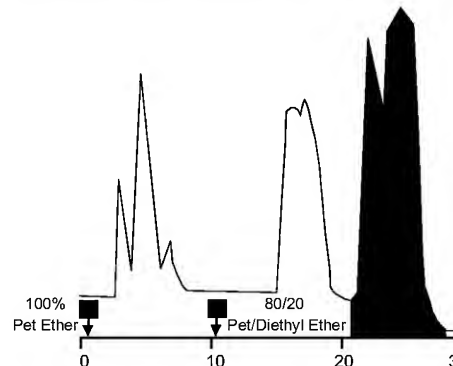
### Tight Particle Size Distribution to Optimize Efficiency and Pressure Drop



\*All comparative data generated on chromatographic silica labeled 60Å, 40–63µm.



## Synthetic Intermediate Purification



**Sample:** 1g Reaction Products  
**Column:** 50 x 500mm  
**Column Packing:** Davisil® LC60Å 20–45µm  
**Mobile Phase:** See Chromatogram  
**Flow Rate:** 175mL/min (535cm/hr)  
**Detection:** UV at 254nm

Davisil® silica purification of a schiff base from a crude reaction mixture containing aldehyde, amine and other by-products. The good separation and loading capacity shown at the pilot scale allowed scale-up to a 300mm diameter column producing over 90g of purified product per run.

## Global Technical Support

To assist customers, Grace offers advice, assistance or laboratory trials for intended process-scale use. Our field representatives can arrange for such support when required, as well as discuss our ability to customize grades tailored to your requirements.



# Davisil® Media

## Greater Selection

A wide range of Davisil® silica grades are available to meet your performance and economic requirements. The selection guide in this catalog will help you choose the best grade for your application.

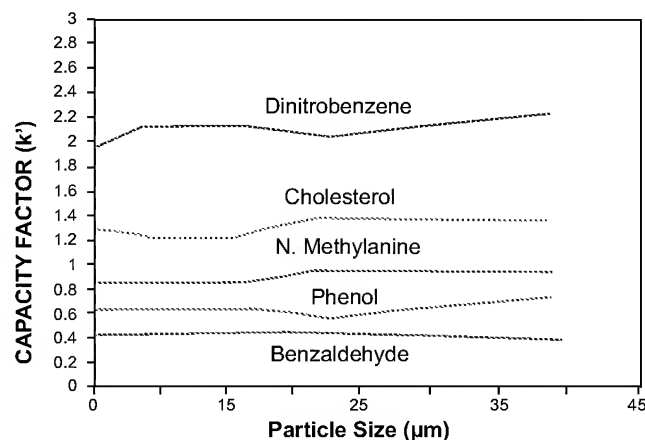
- Available in both normal phase bare silica and various bonding chemistries (C18, Amino, Diol, Cyano) for alternative selectivity
- Wide selection of distinct pore diameters (30–2500Å) for separation of various MW sizes
- Available from 500g to multi-ton quantities



Look for this icon on products from Grace using Davisil® silica, such as TLC plates and SPE/Flash cartridges, to experience the same great performance.

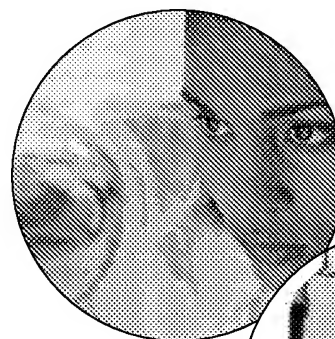
## Predictable Scale-Up

Today we manufacture hundreds of tons of Davisil® chromatographic silica per year in multi-ton lots. Our manufacturing is at scale, so your manufacturing can be at scale. In scaling up, you can be confident that Davisil® chromatographic silica will yield consistent performance as particle size is increased.

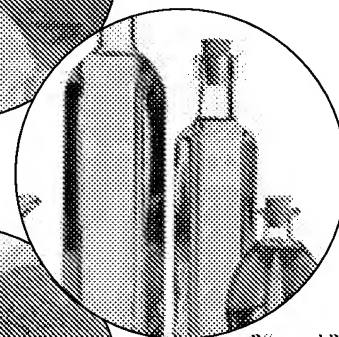


Uniform capacity (k') and selectivity (α) factors across all particle sizes for predictable scale-up.

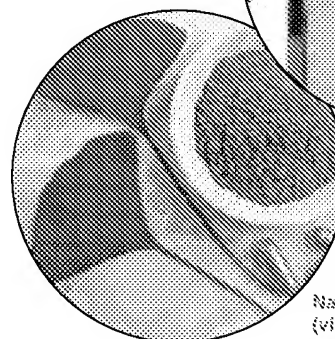
*Davisil® normal-phase silica functions through hydrophilic interactions, with more polar compounds generally retained longer. This makes it ideally suited for purification of:*



Chemical Synthesis Intermediates



Oils and Fats

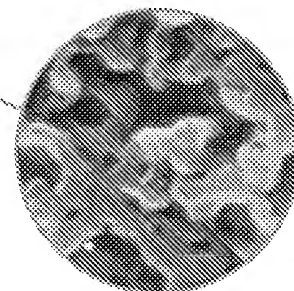


Natural Products (vitamins, flavors, fragrances, etc.)

## Unbeatable Product Reliability

Manufactured for over 25 years, Davisil® chromatographic silica is one of the world's most widely used chromatography sorbents.

Surface Area & Pore Volume:  
±1-10% lot to lot



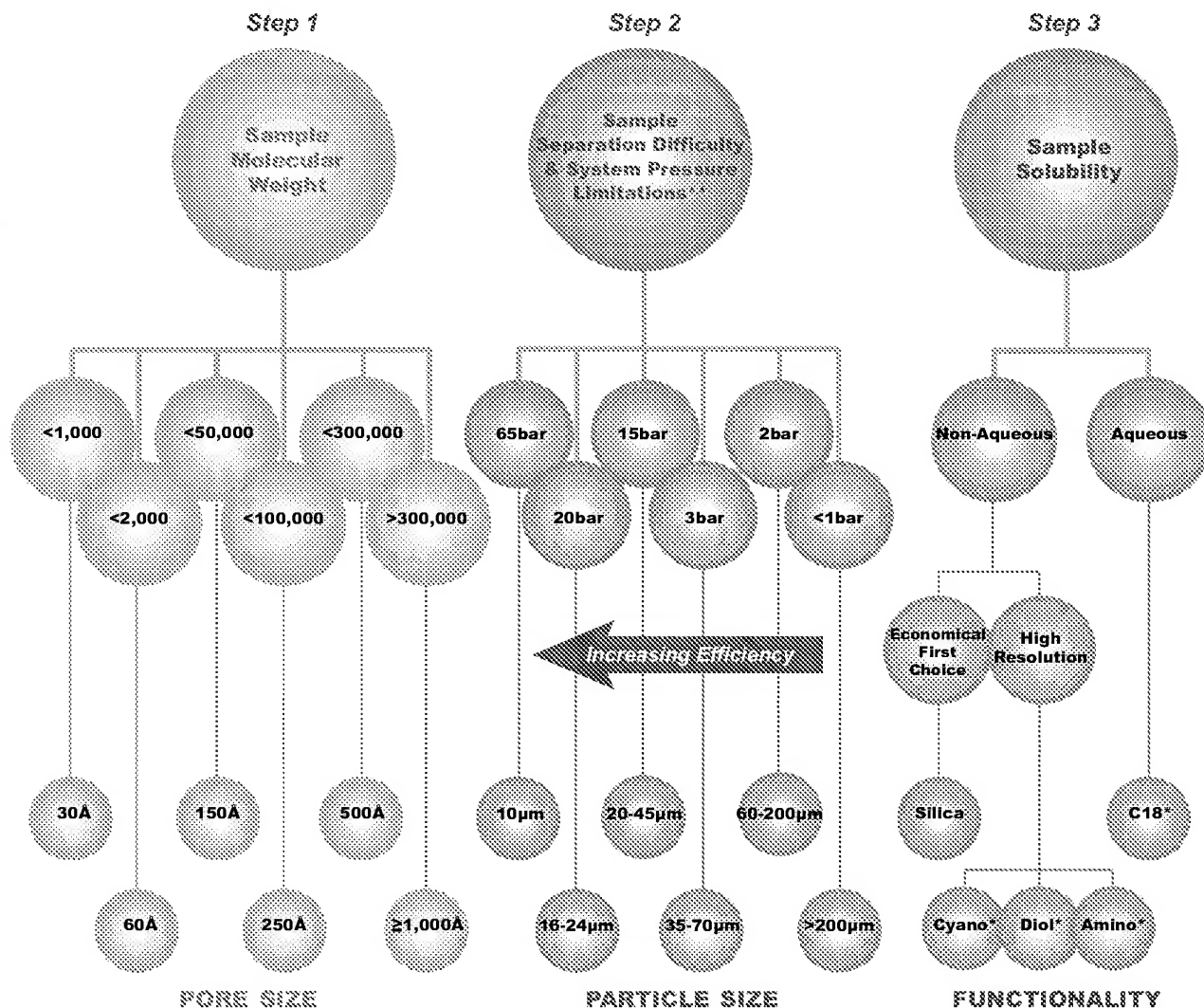
Produced at two ISO-9001 certified facilities under strict QC controls from raw material to finished product insures high lot-to-lot reproducibility.

# Davisil® Media

## Selection Guide

Many types of column packings are suitable for a given application. The diagram below is intended for general guidance to the chromatographer. By following each of the three paths in sequence, the proper packing media is selected.

It should be noted that Grace offers other media to complement the Davisil® packings in many of these applications.



\*\*4.6 x 250mm column, 1mL/min, water (1 cP viscosity).

\*Available on 60Å, 10µm or 50µm particle sizes.

## Typical Physical and Chemical Characteristics

Exact specifications for each grade are available for download at: [www.discoverysciences.com/prep](http://www.discoverysciences.com/prep).

Characteristics	30Å	60Å	150Å	250Å	500Å	1000Å	1500Å	2500Å
Nominal Pore Size	30Å	60Å	150Å	250Å	500Å	1000Å	1500Å	2500Å
Surface Area (m <sup>2</sup> /g)	700	550	330	285	80	40	25	17
Pore Volume (mL/g)	0.4	0.9	1.2	1.8	1.1	1.1	1.1	1.1
pH (5% suspension)	4	7.3	7.3	7.5	8.0	9.0	9.0	9.0
H <sub>2</sub> O (weight %) <sup>†</sup>	<6%	<6%	<6%	<6%	<6%	<6%	<6%	<6%
Bulk Density (kg/m <sup>3</sup> )	720	530	350	210	370	370	370	370

<sup>†</sup>Moisture content (% H<sub>2</sub>O) can be tailored (increased or decreased) to meet customer requirements.

# Davisil® Media

Below is a listing of our more popular Davisil® silica grades.  
For a complete listing request brochure 538.

## Unbonded Silica

APS	Particle Size	Davisil® Silica Grade	Pk Size	Part No.
30A	50–100µm	921	25kg	5101737
			75–150µm	923 <sup>1</sup>
			5lb	5139002
			72 x 10g	5139003
			576 x 10g	5139001
	75–700µm	12 <sup>2</sup>	25kg	5139180
60A	250–500µm	15 <sup>3</sup>	25kg	5101744
	10µm	710NW	20kg	5136220
	16–20µm	631	25kg	5101778
	40–63µm	LC60A 40–63µm	25kg	5054993
			5kg	5098468
			1kg	5134312
	60–200µm	LC60A 60–200µm	25kg	5007446
			5kg	5134295
			1kg	5134311
	200–500µm	LC60A 200–500µm	25kg	5022298
	150A	16–24µm	LC150A 16–24µm	25kg
35–70µm		LC150A 35–70µm	25kg	5057993
			5kg	5134294
			1kg	5134299
60–200µm		LC150A 60–200µm	25kg	5134770
			5kg	5134293
	1kg		5134298	
250A	40–63µm	LC250A 40–63µm	15kg	5134301
			2.5kg	5134292
	60–200µm	LC250A 60–200µm	15kg	5134300
500A	35–70µm	XWP500A 35–70µm	2.5kg	5134291
			20kg	5030057
			5kg	5108711
	90–130µm	XWP500A 90–130µm	20kg	5058842
			5kg	5134305
1000A	100–300µm	XWP500A 100–300µm	20kg	5057050
	16–24µm	XWP1000A 16–24µm	20kg	5134302
			5kg	5016756
			20kg	5034754
	35–70µm	XWP1000A 35–70µm	5kg	5134304
1500A				
20kg			5093501	
1500A	16–24µm	XWP1500A 16–24µm	18kg	5070159
	90–130µm	XWP1500A 90–130µm	18kg	5045916
2500A	90–130µm	XWP2500A 90–130µm	5kg	5107451

## ASTM or EPA Method

<sup>1</sup>Grade 923 ASTM D1319: Petroleum Products by FIA  
ASTM D2549: Aromatics/Non-Aromatics from Oils  
EPA Method 1664: N-Hexane Extraction Method  
<sup>2</sup>Grade 12 ASTM D2007: Rubber Extender/ Processing Oils  
<sup>3</sup>Grade 15 EPA Method 601/624: Purgeable Halocarbons



7174

## Bonded Silica

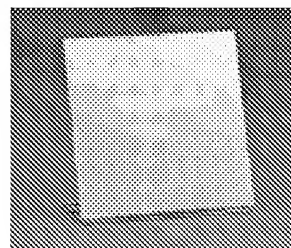
APS	Bonded Phase	Particle Size	Davisil® Silica Grade	Pk Size	Part No.
60A	C18	10µm	710NC18E	250g	5135418
				1kg	5135305
		35–60µm	633NC18E	250g	5135414
				1kg	5134095
	Cyano	10µm	710NCNE	250g	5135419
				1kg	5134223
		35–60µm	633NCNE	250g	5135415
				1kg	5134224
	Diol	10µm	710N2OH	250g	5135417
				1kg	5135303
	Amino	35–60µm	633N2OH	250g	5135413
				1kg	5135302
		10µm	710NNH2	250g	5135420
				1kg	5134682
				250g	5135416
		35–60µm	633NNH2	250g	5135416
				1kg	5134096

## related products

Matching Davisil® Silica TLC plates for easy method development

## Davisil® Silica TLC Plates

Description	Layer Thickness	Qty.	Part No.
<i>Hard Layer, Organic Binder, Fluorescent Indicator, 264nm</i>			
<i>Scored, 4, 5 x 20cm Sections</i>			
20 x 20cm	250µm	25	8617580
<i>Scored, 8, 2.5 x 10cm Sections</i>			
10 x 20cm	250µm	25	8617610



## related products

Column Hardware and Packing  
Equipment also available.  
See pages 162–174.

## related products

Looking for additional TLC products?  
See pages 188–198.

# Nickel(0)-catalyzed asymmetric cross-coupling reactions of allylic compounds with arylboronic acids

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Optically active oxazolinylferrocenylphosphines have been found to work quite effectively as chiral ligands in nickel(0)-catalyzed cross-coupling reactions of allylic compounds with arylboronic acids, which are known to behave as “hard” nucleophiles. The expected coupling products have been obtained in good yields with moderate enantioselectivities (up to 53% ee). This is the first example of asymmetric allylic substitution using organoboron compounds.

## Introduction

Organoboron compounds are well-known as versatile reagents in organic synthesis. A number of studies on transition-metal catalyzed carbon–carbon bond forming reactions using these compounds have recently appeared such as the Suzuki–coupling reaction,<sup>1</sup> conjugate addition to enones,<sup>2</sup> addition to aldehydes,<sup>3</sup> allylic substitution,<sup>4</sup> carbonylation<sup>5</sup> and cross-coupling reaction with alkenes or acid chlorides.<sup>6</sup> Each reaction proceeds *via* either transmetallation between a boron atom and a transition metal or oxidative addition of a carbon–boron bond to a lower valence transition metal. Quite recently, asymmetric versions of these reactions have appeared,<sup>2d,2e,3,7</sup> but to the best of our knowledge there are no reports until now on the catalytic asymmetric allylic substitution using such compounds.

The allylic substitution reaction is a powerful method used to construct a new carbon–carbon bond and, so far, excellent selectivities have been attained using palladium catalysts with various chiral ligands in the allylation with “soft” nucleophiles.<sup>8</sup> However, examples of the reaction with corresponding “hard” nucleophiles are quite limited, most of which are nickel-catalyzed allylic substitutions with Grignard reagents in the presence of chiral P–P ligands.<sup>9</sup> Mechanistically speaking, the allylic substitution reaction with a “hard” nucleophile is considered to proceed differently from that with a “soft” nucleophile; a hard nucleophile attacks a transition metal first (Scheme 1, path (A)), while a “soft” one attacks an allylic carbon directly (Scheme 1, path (B)).<sup>10</sup> In this paper, we disclose

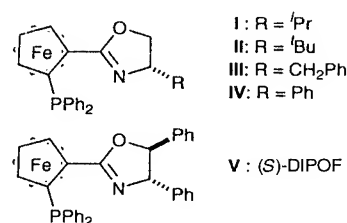
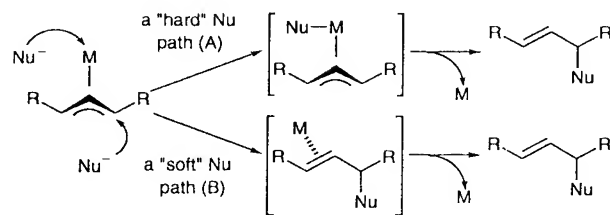


Chart 1

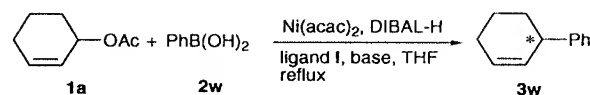
## Results and discussion

3-Acetoxycyclohexene (**1a**) and sodium tetraphenylborate (NaBPh<sub>4</sub>) were chosen first as substrates according to the report by Legros and Fiaud.<sup>4a</sup> The cross-coupling reaction was attempted in tetrahydrofuran (THF) at reflux with **1a** (1 equiv.) and NaBPh<sub>4</sub> (1.5 equiv.) in the presence of nickel(II) acetylacetonate [Ni(acac)<sub>2</sub>] (5 mol%), diisobutylaluminum hydride (DIBAL-H) (16 mol%), and a chiral oxazolinylferrocenylphosphine (**I**, 5 mol%) as a ligand. 3-Phenylcyclohexene (**3w**) was obtained in 28% yield and 1% ee after 42 h, no improvement being shown even by the addition of a base which is indispensable in many cases of Suzuki coupling reactions.<sup>1</sup> Then, hoping to obtain higher chemical yields, we switched from NaBPh<sub>4</sub> to phenylboronic acid (**2w**) (Scheme 2), which is



Scheme 1

that optically active alkenes having an aryl group at the allylic position can be obtained stereoselectively in nickel(0)-catalyzed asymmetric cross-coupling reactions of allylic compounds with “hard” nucleophiles, arylboronic acids, in the presence of a chiral oxazolinylferrocenylphosphine (Chart 1).<sup>11</sup> These ligands have been found to work quite effectively in rhodium(I)-, iridium(I)- or ruthenium(II)-catalyzed hydrosilylation of ketones or imines<sup>12</sup> and ruthenium(II)-catalyzed transfer hydrogenation of ketones.<sup>13</sup>



Scheme 2

usually employed in Suzuki coupling reactions. In the presence of base, the nucleophilicity of an organic group on the boron atom is considered to be enhanced by forming “ate” complexes. As summarized in Table 1, the yield of **3w** was greatly increased. In each case a very small amount of the by-product, biphenyl, was detected on GLC analysis (below 3%). Many inorganic bases such as K<sub>3</sub>PO<sub>4</sub>·*n*H<sub>2</sub>O and KOH significantly enhanced the chemical yields of **3w**, with **3w** scarcely being obtained in the absence of base as expected (entry 1). In order to obtain a higher stereoselectivity it was necessary to use KOH and further investigation showed that the optical yield was dependent on the concentration of the substrate as well as the nickel:ligand ratio. Thus, the desired product was obtained more stereoselectively (50% ee, entry 9) by using a chiral ligand and nickel in a ratio of 2:1 with a higher concentration of **1a**

**Table 1** Effect of base on nickel-catalyzed asymmetric allylic substitution<sup>a</sup>

Entry	Base	Time/h	Yield (%) <sup>b</sup>	Ee (%) <sup>c</sup>
1	None	17	3	— <sup>d</sup>
2	Et <sub>3</sub> N	65	Trace	— <sup>d</sup>
3	K <sub>3</sub> PO <sub>4</sub> ·nH <sub>2</sub> O	17	87	4
4	KO <sup>t</sup> Bu	17	32	4
5	Ca(OH) <sub>2</sub>	118	22	1
6	NaOH	17	57	8
7	KOH	17	65	21
8 <sup>e</sup>	KOH	17	46	28
9 <sup>e,f</sup>	KOH	17	81	50

<sup>a</sup> Reaction conditions; **1a** (0.50 mmol), **2w** (1.5 mmol), Ni(acac)<sub>2</sub> (0.025 mmol), DIBAL-H (0.080 mmol), ligand **I** (0.026 mmol, Ni: ligand = 1:1), base (1.5 mmol), THF (2.5 mL, 0.20 M of **1a**), at reflux. <sup>b</sup> Determined by GLC. <sup>c</sup> Determined by optical rotation; (*S*)-configuration predominated in either case. <sup>d</sup> Not determined. <sup>e</sup> THF (1.5 mL, 0.33 M of **1a**). <sup>f</sup> Ligand **I** (0.050 mmol, Ni:ligand = 1:2).

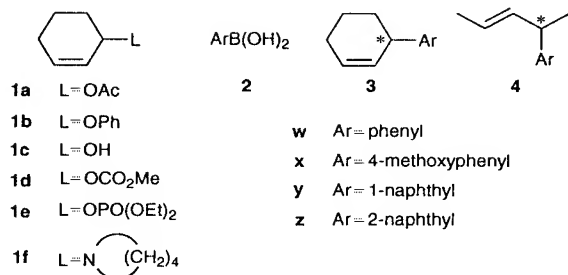
**Table 2** Effect of chiral ligand on nickel-catalyzed asymmetric allylic substitution<sup>a</sup>

Entry	Ligand	Yield (%) <sup>b</sup>	Ee (%) <sup>c</sup>
1	None	2	— <sup>d</sup>
2	<b>I</b>	81	50
3	<b>II</b>	55	9
4	<b>III</b>	56	53
5	<b>IV</b>	20	48
6	<b>V</b>	41	48

<sup>a</sup> Reaction conditions; **1a** (0.50 mmol), **2w** (1.5 mmol), Ni(acac)<sub>2</sub> (0.025 mmol), DIBAL-H (0.080 mmol), ligand (0.050 mmol), KOH (1.5 mmol), THF (1.5 mL, 0.33 M of **1a**), reflux for 17 h. <sup>b</sup> Determined by GLC. <sup>c</sup> Determined by optical rotation; (*S*)-configuration predominated in either case. <sup>d</sup> Not determined.

(0.33 M instead of 0.20 M). In this reaction system, heating to reflux temperature was essential, otherwise **3w** could not be detected at all in GLC analysis (at rt or even at 50 °C). In addition, **3w** was scarcely obtained either by the use of Pd(dba)<sub>2</sub> or Pt(dba)<sub>2</sub> in place of Ni(acac)<sub>2</sub> and DIBAL-H,<sup>14</sup> or by the use of some other organoboron and organoheteroatom compounds such as Ph<sub>3</sub>B-KOH, phenylboronic acid ethylene glycol ester [PhB(OCH<sub>2</sub>CH<sub>2</sub>O)]-KOH or MeLi, Ph<sub>3</sub>Sb-KOH, Ph<sub>3</sub>Bi-KOH and PhSnBu<sub>3</sub>.<sup>15,16</sup>

Next, various other chiral oxazolinylferrocenylphosphines (Chart 1) were used for this reaction (Table 2). Similar enantioselectivities were observed with most ligands including **V** (*S,S,S*)-[2-(4,5-diphenyl-4,5-dihydro-1,3-oxazol-2-yl)ferrocenyl]-diphenylphosphine (abbreviated as (*S*)-**DIPOF**), which is the best ligand in rhodium(i)- and iridium(i)-catalyzed hydrosilylation of ketones,<sup>12a,b</sup> although the use of **II** resulted in lower enantioselectivity (entry 3). As to the effect of the nature of the leaving group of cyclohexenyl compounds (Chart 2), the acetoxy group gave the best result both in chemical and optical yields (Table 3, entry 1), followed by the hydroxy group (entry

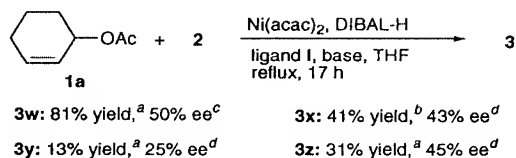
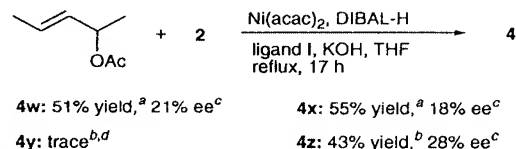
**Chart 2****Table 3** Effect of leaving group on nickel-catalyzed asymmetric allylic substitution<sup>a</sup>

Entry	Y	Yield (%) <sup>b</sup>	Ee (%) <sup>c</sup>
1	<b>1a</b>	81	50
2	<b>1b</b>	8	— <sup>d</sup>
3	<b>1c</b>	50	32
4	<b>1d</b>	7	— <sup>d</sup>
5	<b>1e</b>	6	— <sup>d</sup>
6	<b>1f</b>	34	37

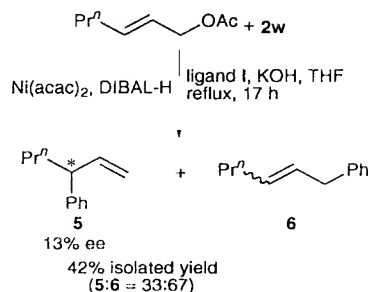
<sup>a</sup> Reaction conditions; **1** (0.50 mmol), **2w** (1.5 mmol), Ni(acac)<sub>2</sub> (0.025 mmol), DIBAL-H (0.080 mmol), ligand **I** (0.050 mmol), KOH (1.5 mmol), THF (solvent, 0.33 M of an allylic substrate), at reflux for 17 h. <sup>b</sup> Determined by GLC. <sup>c</sup> Determined by optical rotation; (*S*)-configuration predominated in either case. <sup>d</sup> Not determined.

3) and pyrrolidinyl group employed by Trost and Spagnol<sup>4b</sup> (entry 6). In the absence of KOH, compound **1f** hardly reacted with **2w**, although this type of amine is known to work as a base as well.<sup>4b</sup>

Using the conditions described above, the reactions of other arylboronic acids (Chart 2) with **1a** (Scheme 3) and an acyclic substrate, 4-acetoxypent-2-ene (Scheme 4) were carried out. In

**Scheme 3** <sup>a</sup> GLC yield. <sup>b</sup> Isolated yield. <sup>c</sup> Determined by optical rotation. <sup>d</sup> Determined by HPLC using a suitable chiral column.**Scheme 4** <sup>a</sup> Isolated yield. <sup>b</sup> GLC yield. <sup>c</sup> Determined by HPLC using a suitable chiral column. <sup>d</sup> The ee value was not determined.

all cases the expected coupling products were obtained, but both the chemical yield and enantioselectivity were lower, unfortunately. An unsymmetrical substrate, 1-acetoxyhex-2-ene, reacted with **2w** to afford the coupling products **5** and **6** (Scheme 5), albeit the selectivity for **5** as well as its enantioselectivity was not high (**5**:**6** = 33:67, 13% ee).<sup>17,18</sup>

**Scheme 5**

In conclusion, we have found that optically active oxazolinylferrocenylphosphines work as chiral ligands in nickel(0)-catalyzed cross-coupling reactions of allylic compounds with arylboronic acids to afford the desired arylated products with moderate enantioselectivities (up to 53% ee).



## Experimental

### General

<sup>1</sup>H- and <sup>13</sup>C-NMR spectra were measured on JEOL EX-400, JEOL JNM-AL300 and JEOL JNM-GSX270 spectrometers for solutions in CDCl<sub>3</sub> with Me<sub>4</sub>Si as an internal standard. GLC analyses were carried out with a Shimadzu GC-14A instrument equipped with a CPB 10-S25-050 column (Shimadzu, fused silica capillary column, 0.33 mm × 25 m, 5.0 mm film thickness) using helium as carrier gas. GLC yields were determined using bibenzyl as an internal standard. Optical rotations were measured on a JASCO DIP-1000 instrument. HPLC analyses were carried out on an HLC-803A instrument (Tosoh) with a UV-8011 detector using a Daicel Chiralcel OB, OD or OJ column. [α]<sub>D</sub> values are measured in 10<sup>-1</sup> deg cm<sup>2</sup> g<sup>-1</sup>. Analytical thin layer chromatography (TLC) was performed with silica gel 60 Merck F-254 plates. Column chromatography was performed with Merck silica gel 60.

### Materials

Tetrahydrofuran (THF) was distilled from sodium benzophenone ketyl under argon. Triethylamine was distilled from calcium hydride. Other commercially available organic and inorganic compounds including Ni(acac)<sub>2</sub> and DIBAL-H (diisobutylaluminium hydride) were used without further purification. Chiral oxazolonylferrocenylphosphines (**I–V**) were prepared by reported methods.<sup>11</sup> Cyclohex-2-enol (**1c**) was prepared by the reduction of cyclohex-2-en-1-one with NaBH<sub>4</sub> and CeCl<sub>3</sub>·7H<sub>2</sub>O in methanol.<sup>19</sup> 3-Acetoxycyclohexene (**1a**) and 1-acetoxycyclohex-2-ene were prepared from the corresponding alcohol and acetic anhydride. Cyclohex-2-enyl carbonate (**1d**) was prepared from **1c** and methyl chlorocarbonate. Cyclohex-2-enyl diethyl phosphate (**1e**) was prepared from **1c** and chlorophosphoric acid diethyl ester. 3-Phenoxycyclohexene (**1b**),<sup>20</sup> 3-pyrrolidin-1-ylcyclohexene (**1f**),<sup>46</sup> 4-acetoxypent-2-ene<sup>21</sup> and arylboronic acids (**3x**, **3y**, **3z**)<sup>22</sup> as well as Pd(dba)<sub>2</sub><sup>23</sup> and Pt(dba)<sub>2</sub><sup>24</sup> were prepared according to literature procedures.

### General procedure for Ni(0)-catalyzed cross-coupling reaction of 3-acetoxycyclohexene (**1a**) with phenylboronic acid (**2w**) (Table 1, entry 9)

A mixture of Ni(acac)<sub>2</sub> (6.5 mg, 0.025 mmol), (*S,S*)-[2-(4-isopropyl-4,5-dihydro-1,3-oxazol-2-yl)ferrocenyl]diphenylphosphine (**I**, 24.2 mg, 0.050 mmol) and bibenzyl (as an internal standard; 19.9 mg) was stirred for 1 h in THF (0.5 mL) at room temperature under nitrogen. After cooling to 0 °C, DIBAL-H (1.0 M solution in hexane; 0.08 mL, 0.08 mmol) was added to the mixture which was stirred for another 30 min. A solution of 3-acetoxycyclohexene (**1a**, 70.0 mg, 0.50 mmol) in THF (0.5 mL), phenylboronic acid (**2w**, 183 mg, 1.5 mmol), potassium hydroxide (85.5 mg, 1.5 mmol) and THF (0.5 mL) were added successively to the mixture and the resulting mixture was heated under reflux for 17 h. The resulting mixture was diluted with hexane and filtered through Florisil. The amount of product **3w** was determined by GLC analysis. For isolation of **3w** the solvent was evaporated and the residue was purified by column chromatography using hexane as an eluent. Typical spectroscopic data of the obtained coupling products are as follows.

**3-Phenylcyclohexene (3w).** A colorless liquid; <sup>1</sup>H-NMR δ = 1.49–2.11 (6H, m), 3.42 (1H, m), 5.71 (1H, m), 5.89 (1H, m), 7.16–7.33 (5H, m); <sup>13</sup>C-NMR δ = 21.19, 25.01, 32.60, 41.85, 125.94, 127.71, 128.24, 128.33, 130.18, 146.64. The ee value and the configuration of the product were determined by a polarimeter based on the reported rotation of an optically pure (*R*)-**3w**, [α]<sub>D</sub><sup>29</sup> = +159.6 (c 0.53, benzene).<sup>25</sup>

**3-(4-Methoxyphenyl)cyclohexene (3x).** A colorless liquid; <sup>1</sup>H-NMR δ = 1.48–1.67 (3H, m), 1.69–1.77 (1H, m), 1.95–2.01

(1H, m), 2.06–2.10 (2H, m), 3.32–3.38 (1H, m), 3.79 (3H, s), 5.69 (1H, dd, *J* = 10.0, 2.2 Hz), 5.83–5.89 (1H, m), 6.84 (2H, d, *J* = 8.5 Hz), 7.13 (2H, d, *J* = 8.5 Hz); <sup>13</sup>C-NMR δ = 21.13, 25.03, 32.73, 40.97, 55.27, 113.69, 128.13, 128.61, 130.53, 138.81, 157.89. The ee value was determined by HPLC analysis with a Daicel Chiralcel OB column using hexane as an eluent.

**3-(1-Naphthyl)cyclohexene (3y).** A colorless liquid; <sup>1</sup>H-NMR δ = 1.64–1.78 (3H, m), 2.15–2.21 (3H, m), 4.19–4.26 (1H, m), 5.83 (1H, dd, *J* = 10.0, 2.6 Hz), 5.98–6.05 (1H, m), 7.37–7.54 (4H, m), 7.72 (1H, dd, *J* = 7.4, 2.1 Hz), 7.87 (1H, dd, *J* = 7.4, 2.2 Hz), 8.13 (1H, d, *J* = 7.7 Hz); <sup>13</sup>C-NMR δ = 20.86, 25.25, 30.91, 37.00, 123.41, 125.06, 125.27, 125.42, 125.71, 126.60, 126.77, 128.92, 130.22, 131.40, 134.10, 141.92; IR (neat) 724, 761, 778, 796, 2834, 2858, 2930, 3018, 3045, 3059 cm<sup>-1</sup> (Anal. Calcd. for C<sub>16</sub>H<sub>16</sub>: C, 92.26; H, 7.74. Found: C, 92.50; H, 7.83%). The ee value was determined by HPLC analysis with a Daicel Chiralcel OB column using hexane as an eluent.

**3-(2-Naphthyl)cyclohexene (3z).** A colorless liquid; <sup>1</sup>H-NMR δ = 1.62–2.23 (6H, m), 3.56–3.60 (1H, m), 5.81 (1H, dd, *J* = 10.0, 2.1 Hz), 5.92–6.00 (1H, m), 7.35–7.61 (3H, m), 7.64 (1H, s), 7.76–7.88 (3H, m); <sup>13</sup>C-NMR δ = 21.12, 25.09, 32.41, 41.91, 125.14, 125.79, 125.81, 126.71, 127.55, 127.58, 127.84, 128.64, 130.06, 132.16, 133.55, 144.06; IR (neat) 723, 744, 757, 815, 853, 2835, 2856, 2927, 3018, 3052 cm<sup>-1</sup> (Anal. Calcd. for C<sub>16</sub>H<sub>16</sub>: C, 92.26; H, 7.74. Found: C, 92.45; H, 7.70%). The ee value was determined by HPLC analysis with a Daicel Chiralcel OJ column using hexane as an eluent.

**4-Phenylpent-2-ene (4w).** A colorless liquid; <sup>1</sup>H-NMR δ = 1.33 (3H, d, *J* = 7.3 Hz), 1.67 (3H, d, *J* = 7.3 Hz), 3.41 (1H, m, *trans* isomer), 3.79 (1H, m, *cis* isomer), 5.42–5.65 (2H, m), 7.16–7.31 (5H, m); <sup>13</sup>C-NMR δ = 17.88, 21.46, 42.33, 123.63, 125.90, 127.13, 128.32, 136.24, 146.48. The diastereomeric ratio of **4w** (*trans*:*cis* = 92:8) was determined by <sup>1</sup>H-NMR analysis and the ee value was determined by HPLC analysis with a Daicel Chiralcel OJ column using hexane as an eluent.

**4-(4-Methoxyphenyl)pent-2-ene (4x).** A colorless liquid; <sup>1</sup>H-NMR δ = 1.30 (3H, d, *J* = 7.0 Hz), 1.66 (3H, d, *J* = 6.1 Hz), 3.36 (1H, m, *trans* isomer), 3.76 (1H, m, *cis* isomer), 3.78 (3H, s), 5.39–5.49 (1H, m), 5.55–5.62 (1H, m), 6.84 (2H, d, *J* = 8.7 Hz), 7.12 (2H, d, *J* = 8.7 Hz); <sup>13</sup>C-NMR δ = 17.88, 21.57, 41.45, 55.25, 113.73, 123.33, 128.00, 136.58, 138.62, 157.79. The diastereomeric ratio was not determined because the peak of the allylic proton of the *cis* isomer overlaps with that of the methyl protons of the methoxy group in the <sup>1</sup>H-NMR spectrum. The ee value was determined by HPLC analysis with a Daicel Chiralcel OJ column using hexane as an eluent.

**4-(1-Naphthyl)pent-2-ene (4y).** A colorless liquid; <sup>1</sup>H-NMR δ = 1.47 (3H, d, *J* = 6.8 Hz, *trans* isomer), 1.48 (3H, d, *J* = 7.0 Hz, *cis* isomer), 1.69 (3H, dt, *J* = 6.4, 1.4 Hz, *trans* isomer), 1.74 (3H, dd, *J* = 6.6 Hz, *cis* isomer), 4.24 (1H, m, *trans* isomer), 4.53 (1H, m, *cis* isomer), 5.46–5.80 (2H, m), 7.36–7.53 (4H, m), 7.70 (1H, d, *J* = 7.7 Hz), 7.84 (1H, m), 8.13 (1H, d, *J* = 7.7 Hz); <sup>13</sup>C-NMR δ = 17.98, 21.06, 37.02, 123.43, 123.61, 124.13, 125.25, 125.58, 125.63, 126.56, 128.84, 131.44, 133.96, 135.82, 142.34.

**4-(2-Naphthyl)pent-2-ene (4z).** A colorless liquid; <sup>1</sup>H-NMR δ = 1.42 (3H, d, *J* = 6.9 Hz), 1.69 (3H, dt, *J* = 6.0, 1.2 Hz, *trans* isomer), 1.73 (3H, d, *J* = 1.4 Hz, *cis* isomer), 3.53–3.63 (1H, m, *trans* isomer), 3.89–4.01 (1H, m, *cis* isomer), 5.44–5.57 (1H, m), 5.65–5.74 (1H, ddq, *J* = 15.3, 6.6, 1.4 Hz), 7.36 (1H, dd, *J* = 8.5, 1.9 Hz), 7.40–7.47 (2H, m), 7.62 (1H, s), 7.75–7.81 (3H, m); <sup>13</sup>C-NMR δ = 17.94, 21.37, 42.41, 124.01, 124.93, 125.15, 125.81, 126.31, 127.55, 127.60, 127.84, 132.14, 133.64, 136.10, 143.91. The diastereomeric ratio of **4z** (*trans*:*cis* = 92:8) was

determined by  $^1\text{H}$ -NMR analysis and the ee value was determined by HPLC analysis with a Daicel Chiralcel OD column using hexane as an eluent.

**3-Phenylhex-1-ene (5).** A colorless liquid;  $^1\text{H}$ -NMR  $\delta$  = 0.89 (3H, t,  $J$  = 7.8 Hz), 1.16–1.42 (2H, m), 1.65–1.71 (2H, m), 3.25 (1H, dt,  $J$  = 7.6, 7.6 Hz), 5.01 (1H, d,  $J$  = 13.9 Hz), 5.03 (1H, d,  $J$  = 8.9 Hz), 5.95 (1H, ddd,  $J$  = 13.9, 8.9, 7.6 Hz), 7.17–7.19 (3H, m), 7.26–7.31 (2H, m);  $^{13}\text{C}$ -NMR  $\delta$  = 13.98, 20.62, 37.63, 49.62, 113.80, 126.05, 127.60, 128.39, 142.53, 144.67. The ee value was determined by HPLC analysis with a Daicel Chiralcel OJ column using hexane as an eluent.



**1-Phenylhex-2-ene (6).** A colorless liquid;  $^1\text{H}$ -NMR  $\delta$  = 0.90 (3H, t,  $J$  = 7.3 Hz), 1.35–1.44 (2H, m), 2.00 (2H, dt,  $J$  = 6.8, 7.3 Hz, *trans* isomer), 2.14 (2H, dt,  $J$  = 7.1, 7.3 Hz, *cis* isomer), 3.33 (2H, d,  $J$  = 6.3 Hz, *trans* isomer), 3.40 (2H, d,  $J$  = 6.4 Hz, *cis* isomer), 5.47–5.61 (2H, m), 7.17–7.19 (3H, m), 7.25–7.30 (2H, m);  $^{13}\text{C}$ -NMR  $\delta$  = 13.67, 22.59, 34.59, 39.05, 125.83, 128.30, 128.46, 128.88, 131.88, 141.14. The diastereomeric ratio of 6 (*trans*:*cis* = 90:10) was determined by  $^1\text{H}$ -NMR analysis.

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- The yield of compound **3w** was as follows: trace(Pd(dba)<sub>2</sub>):ligand **I** = 1:1), 2%(Pd(dba)<sub>2</sub>):ligand **I** = 1:2), 5%(Pt(dba)<sub>2</sub>):ligand **I** = 1:1).
- 3-Bromocyclohexene was used instead of **1a** in the reaction with PhSnBu<sub>3</sub>.
- In all cases, the yield of compound **3w** was below 6%.
- There are examples in which the product having a chiral center was obtained predominantly with good enantioselectivity in the reaction using an unsymmetrical substrate and a Grignard reagent; see refs. **9a** and **18**.
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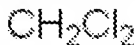
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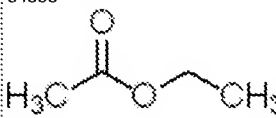
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## Dichloromethane

CHROMASOLV®, for HPLC,  
 ≥99.8%, contains amylene as  
 stabilizer

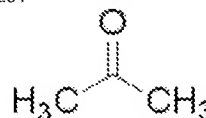
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## Ethyl acetate

CHROMASOLV®, for HPLC,  
 ≥99.7%

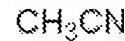
24201



## Acetone

puriss., meets analytical  
 specification of Ph. Eur., BP, NF,  
 ≥99% (GC)

34851



## Acetonitrile

CHROMASOLV® gradient grade,  
 for HPLC, ≥99.9%

Details

Related Products

References

Reviews

## Description

**Application** Suitable for low- and medium-pressure liquid chromatography.

**Packaging** 1 kg in poly btl  
 100 g in poly btl  
 25 kg in fiber drum  
 5 kg in poly drum  
 25 kg, 5 kg packaged in open-head poly drums; 100 g, 1 kg packaged in poly bottles

**Legal Information** Merck is a registered trademark of Merck & Co., Inc.

## Properties

**grade** Grade 9385  
**mfr. no.** Merck®  
**particle size** 230-400 mesh  
 40-63 µm  
**pore size** ~0.8 cm<sup>3</sup>/g pore volume  
 60 Å  
**surface area** 550 m<sup>2</sup>/g  
**bp** 2230 °C  
**mp** >1600 °C

## Safety

**Safety Statements** 22-24/25  
**WGK Germany** NWG  
**RTECS** VV7315000

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# ChromBook 2008|09

Chromatography at Merck –  
Experience drives Innovation



# Standardised silica gels

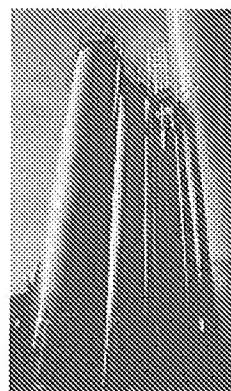
Standardised silica gels from Merck are produced using a traditional method in the world's largest and most modern production plant.

Standardised silica gels are widely used in separation processes to purify high value compounds in ton quantities.

Standardised silica gels are available in many particle size ranges all derived from a single base silica gel, produced only for chromatography.

Standardised silica gels offer easy development of your process from thin layer chromatography to any scale.

Standardised silica gels are available in a wide variety of pack sizes from 500 g to 400 kg to suit your specific needs. Typically, bottles and drums out of pure HDPE are used that are approved for pharmaceutical and food applications.



Typical technical data of standardised silica gel packing materials

Packing material	Characteristics	Spec. surface area $S_{BET}$ ( $m^2/g$ )	Pore volume $V_p$ (ml/g)	pH*	Water content (%)
Silica gel 40	irregular particles of silica; mean pore size: 4 nm (40Å)	600	0.6	7.0	< 7
Silica gel 60	irregular particles of silica; mean pore size: 6 nm (60Å)	500	0.8	7.0	< 7
Silica gel 100	irregular particles of silica; mean pore size: 10 nm (100Å)	360	0.8	7.0	< 7

\*pH of 10 % aqueous suspension

# Standardised silica gels

## Ordering information of silica gel packing materials

Product	Ordering No.	Particle size	Contents
Silica gel 40	1.10180.1000	63 - 200 µm (70 - 230 mesh ASTM)	1 kg
Silica gel 40	1.10180.5000	63 - 200 µm (70 - 230 mesh ASTM)	5 kg
Silica gel 40	1.10180.9025	63 - 200 µm (70 - 230 mesh ASTM)	25 kg
Silica gel 40	1.10181.1000	200 - 500 µm (35 - 70 mesh ASTM)	1 kg
Silica gel 40	1.10181.5025	200 - 500 µm (35 - 70 mesh ASTM)	25 kg
Silica gel 60	1.15111.1000	15 - 40 µm	1 kg
Silica gel 60	1.15111.2500	15 - 40 µm	2.5 kg
Silica gel 60	1.15111.9025	15 - 40 µm	25 kg
Silica gel 60	1.09389.5000	35 - 70 µm (200 - 400 mesh ASTM)	5 kg
Silica gel 60	1.09389.9025	35 - 70 µm (200 - 400 mesh ASTM)	25 kg
Silica gel 60	1.09385.1000	40 - 63 µm (230 - 400 mesh ASTM)	1 kg
Silica gel 60	1.09385.2500	40 - 63 µm (230 - 400 mesh ASTM)	2.5 kg
Silica gel 60	1.09385.5000	40 - 63 µm (230 - 400 mesh ASTM)	5 kg
Silica gel 60	1.09385.9025	40 - 63 µm (230 - 400 mesh ASTM)	25 kg
Silica gel 60	1.07729.1000	< 63 µm (> 230 mesh ASTM)	1 kg
Silica gel 60	1.07729.5000	< 63 µm (> 230 mesh ASTM)	5 kg
Silica gel 60	1.07729.9025	< 63 µm (> 230 mesh ASTM)	25 kg
Silica gel 60	1.15101.1000	63 - 100 µm (170 - 230 mesh ASTM)	1 kg
Silica gel 60	1.15101.9025	63 - 100 µm (170 - 230 mesh ASTM)	25 kg
Silica gel 60	1.07734.1000	63 - 200 µm (70 - 230 mesh ASTM)	1 kg
Silica gel 60	1.07734.2500	63 - 200 µm (70 - 230 mesh ASTM)	2.5 kg
Silica gel 60	1.07734.5000	63 - 200 µm (70 - 230 mesh ASTM)	5 kg
Silica gel 60	1.07734.9025	63 - 200 µm (70 - 230 mesh ASTM)	25 kg
Silica gel 60 extra pure	1.07754.0500	63 - 200 µm (70 - 230 mesh ASTM)	500 g
Silica gel 60 extra pure	1.07754.1000	63 - 200 µm (70 - 230 mesh ASTM)	1 kg
Silica gel 60	1.07733.0500	200 - 500 µm (35 - 70 mesh ASTM)	500 g
Silica gel 60	1.07733.1000	200 - 500 µm (35 - 70 mesh ASTM)	1 kg
Silica gel 60	1.07733.5000	200 - 500 µm (35 - 70 mesh ASTM)	5 kg
Silica gel 60	1.07733.9025	200 - 500 µm (35 - 70 mesh ASTM)	25 kg
Silica gel 100	1.10184.0500	63 - 200 µm (70 - 230 mesh ASTM)	500 g
Silica gel 100	1.10184.5000	63 - 200 µm (70 - 230 mesh ASTM)	5 kg
Silica gel 100	1.10184.9025	63 - 200 µm (70 - 230 mesh ASTM)	25 kg
Silica gel 100	1.10185.0500	200 - 500 µm (35 - 70 mesh ASTM)	500 g
Silica gel 100	1.10185.9025	200 - 500 µm (35 - 70 mesh ASTM)	25 kg
Silica gel 60 F <sub>254</sub> adjusted to 40 % rel. humidity suitable for dry column chromatography	1.10757.1000	63 - 200 µm (70 - 230 mesh ASTM)	1 kg